

December 23, 2016

Defense Technical Information Center  
8725 John J Kingman Road, Suite 0944  
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Reference: N00014-13-1-0274  
PI Name: Kwang Kim, Ph. D.  
University of Nevada, Las Vegas

Subject: Final Report(s)

To whom it may concern:

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- Final Technical Report with SF298

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Sincerely,



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<b>1. REPORT DATE (DD-MM-YYYY)</b> 12/15/2016		<b>2. REPORT TYPE</b> Final Technical		<b>3. DATES COVERED (From - To)</b> 1/1/2013 - 6/30/2016	
<b>4. TITLE AND SUBTITLE</b> Artificial Muscle (AM) Cilia Array for Underwater Systems				<b>5a. CONTRACT NUMBER</b>	
				<b>5b. GRANT NUMBER</b> N000141310274	
				<b>5c. PROGRAM ELEMENT NUMBER</b>	
<b>6. AUTHOR(S)</b> Kwang J. Kim (PI) and Kam Leang (Co-PI)				<b>5d. PROJECT NUMBER</b>	
				<b>5e. TASK NUMBER</b>	
				<b>5f. WORK UNIT NUMBER</b>	
<b>7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)</b> Board of Regents, OBO University of Nevada, Las Vegas 4505 Maryland Parkway Las Vegas, NV 89154-9900				<b>8. PERFORMING ORGANIZATION REPORT NUMBER</b>	
<b>9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)</b> Office of Naval Research 300 Fifth Avenue, Suite 710 Seattle, WA 98104				<b>10. SPONSOR/MONITOR'S ACRONYM(S)</b>	
				<b>11. SPONSOR/MONITOR'S REPORT NUMBER(S)</b>	
<b>12. DISTRIBUTION/AVAILABILITY STATEMENT</b> Approved for Public Release; Distribution is Unlimited					
<b>13. SUPPLEMENTARY NOTES</b>					
<b>14. ABSTRACT</b> <p>The goal of this project is to exploit the unique properties of a new enabling "Artificial Muscle (AM)" to create a cilia-based system for possible use in aquatic applications. Specifically, this project aims to extend the fundamental science behind IPMC material and through design and fabrication create a cilia-based system. An AM is a multifunctional, smart polymer whose electromechanical properties can be controlled, resulting in reproducible actuation and sensing capabilities. Like biological muscles, the AM technology exhibits large motion, good force, fast response, good efficiency, long cycle life, and silent operation.</p>					
<b>15. SUBJECT TERMS</b>					
<b>16. SECURITY CLASSIFICATION OF:</b>			<b>17. LIMITATION OF ABSTRACT</b>	<b>18. NUMBER OF PAGES</b>	<b>19a. NAME OF RESPONSIBLE PERSON</b>
<b>a. REPORT</b>	<b>b. ABSTRACT</b>	<b>c. THIS PAGE</b>			<b>19b. TELEPHONE NUMBER (Include area code)</b>

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## I. Heading

A. PI Name:	Kwang J. Kim <sup>1</sup> (PI) and Kam Leang <sup>2</sup> (Co-PI)
B. Organization:	University of Nevada, Las Vegas and University of Utah
C. ONR Award Number:	N000141310274
D. Award Title:	Artificial Muscle (AM) Cilia Array for Underwater Systems

## II. Scientific and Technical Objectives

The goal of this project is to exploit the unique properties of a new enabling “Artificial Muscle (AM)” to create a cilia-based system for possible use in aquatic applications. Specifically, this project aims to extend the fundamental science behind IPMC<sup>3</sup> material and through design and fabrication create a cilia-based system. An AM is a multifunctional, smart polymer whose electromechanical properties can be controlled, resulting in reproducible actuation and sensing capabilities. Like biological muscles, the AM technology exhibits large motion, good force, fast response, good efficiency, long cycle life, and silent operation. Recent research on the locomotion of biological cells and microorganisms has identified the importance of cilium (flagellum) and its unique motion for locomotion. However, recent man-made attempts at mimicking the motion of cilium using traditional actuators have yielded limited success and often the designs are bulky. On the other hand, the AM technology is an excellent candidate for realizing the complex oscillatory motion of cilium for the development of efficient and highly maneuverable man-made underwater systems as envisioned by Bandyopadhyay and his coworkers<sup>4</sup>. By developing an array of AM-based cilia, a rigorous investigation and study can be conducted to determine the feasibility, effectiveness, and application of the AM technology for marine systems.

The proposed technical objectives, during the project period of 1 June 2015 through 31 May 2016, include (i) Performance characterization of actuation using image-based approach, (ii) Fabrication of various custom-shaped AM structures via additive manufacturing technique, and (iii) development of AM fiber cilia array.

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<sup>2</sup> Associate Professor, Mechanical Engineering Department, University of Utah, Salt Lake City, Utah 84112 (former at the Univ. of Nevada, Reno, Reno, NV 89557)

<sup>3</sup> Ionic Polymer-Metal Composite (IPMC)

<sup>4</sup> P. R. Bandyopadhyay and J. C. Hansen, “Breakup and then makeup: a predictive model of how cilia self-regulate hardness for posture control,” Scientific Reports, Article number: 1956 (2013).

### III. Approach

**Performance characterization of actuation using image-based approach:** The actuation performance of cilia-like IPMC structures was characterized using an image-based approach.

**Fabrication of various custom-shaped AM structures via additive manufacturing technique:** A new method was developed for fused-filament additive manufacturing (3D printing) of IPMC material to create custom-shaped AM structures, including cilia-like structures. Various custom-shaped AM structures were fabricated via additive manufacturing technique.

**Development of an alternative method of fabricating small-diameter cylindrical-shaped Nafion structures for fabricating IPMC actuators:** A method of extruding cylindrical IPMCs was developed. It does not require the use of a mold, but rather involves extrusion of melt Nafion precursor material through a small-diameter hole.

**Development of square cross-section AM fibers:** Square cross-section AM fibers were developed using an in-house cutting and slicing process.

**Development of AM fiber cilia array:** A prototype cilia array (2x2) was fabricated by integrating square cross-section IPMC actuators with a printed circuit board power delivery system.

### IV. Concise Accomplishments

**Performance characterization of actuation using image-based approach:** The actuation performance of cilia-like IPMC structures was characterized using an image-based approach. The approach was developed to address the challenges of adding displacement sensors to the small cilia-like IPMC structures. The image-based approach is non-contact and non-invasive, giving the ability to capture, measure, and record the tip motion of the IPMC structure in an aqueous environment without affecting the IPMC structure. The image-based approach also provided 2D motion characterization and by adding additional cameras, 3D motion can be captured. The image-based approach utilized a digital video camera. The videos were processed using inRange and circle functions in OpenCV software, where thresholding based on the h value in hsv space were used. The software algorithm tracked the center of ellipse fitted to the endpoint of a sample cilia-like IPMC structure with dimensions of approximately 0.5x0.5x35 mm. The 2D motion characterization demonstrated independent control of the actuation in the x- and y-axis, as well as simultaneous 2D motion control.

**Fabrication of various custom-shaped AM structures via additive manufacturing technique:** A new method was developed for fused-filament additive manufacturing (3D printing) of IPMC material to create custom-shaped AM structures, including cilia-like structures. Specifically, a custom 3D printer was created that utilizes custom-made Nafion filament for 3D printing of custom-shaped AM structures. The feature resolution of the 3D printer is approximately 100  $\mu\text{m}$  and various nozzle diameters can be incorporated. The 3D printer can be used to create small-diameter cilia-like IPMC structures as well as other macro-sized structures. Various custom-shaped AM structures were fabricated via additive manufacturing technique, including a thin membrane-like actuator and a linear actuator, and the performance of the devices were demonstrated.



**Development of an alternative method of fabricating cylindrical-shaped IPMC actuators:** A method of fabricating cylindrical IPMCs was developed that involves hot extrusion through a die, where Nafion structures with diameter of 0.5 mm were created. Combining the process with melt-drawing technique produced cylindrical Nafion fibers with diameter size between 180–300  $\mu\text{m}$ , depending on pulling speed.

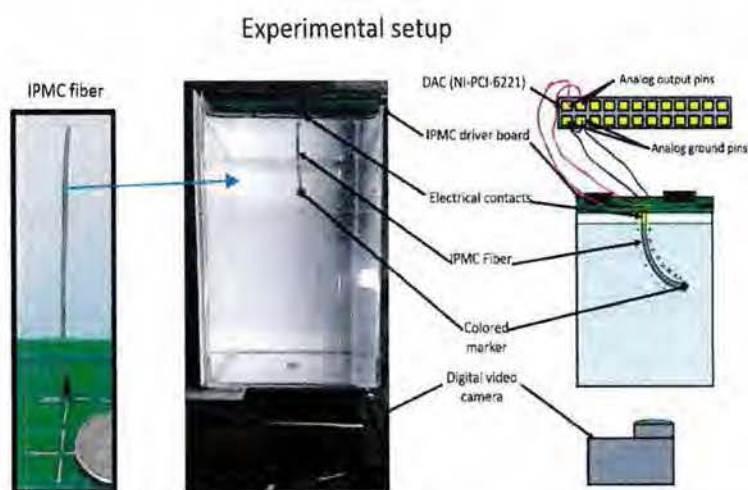
**Development of square cross-section IPMC Actuators:** Square cross-section AM fibers were developed using an in-house cutting process, that produced fibers with 135  $\mu\text{m}$  and 500  $\mu\text{m}$  square cross-sections which were subsequently plated. Smaller square cross-section Nafion fibers with thickness of 54  $\mu\text{m}$  were created using a special slicing technique.

**Development of AM fiber cilia array:** A prototype cilia array was fabricated by integrating square cross-section IPMC actuators with printed circuit board power delivery system. The board has gold plated “L” shaped contacts for gripping the actuators and delivering voltage to drive each IPMC cilia independently in two directions.

## V. Expanded Accomplishments

**Performance characterization of actuation using image-based approach:** The actuation performance of cilia-like IPMC structures was characterized using an image-based approach. The approach was developed to address the challenges of adding displacement sensors to the small cilia-like IPMC structures. It is pointed out that non-contact and non-invasive performance characterization is a preferred method to avoid interactions that can affect the performance. Thus, the image-based approach gives the ability to capture, measure, and record the tip motion of the IPMC structure in an aqueous environment without affecting the IPMC structure. The image-based approach also provided 2D motion characterization and by adding additional cameras, 3D motion can be captured.

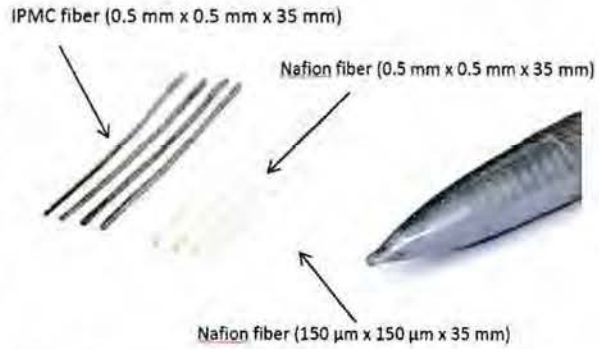
Figure 1 shows the experimental setup for image-based characterization of cilia-like IPMC actuator. The actuator shown can be controlled in two lateral directions, x and y, with two independent inputs. The dimensions of the cilia-like actuator is 0.5x0.5x35 mm, similar the examples shown in Figure 2. As shown, the setup consists of an 2D IPMC actuator with colored marker attached to the free end. A digital video camera is used to record the motion of the IPMC actuator and marker. A custom-designed voltage amplifier is used to apply voltages to the electrodes. The videos were processed using inRange and circle functions in OpenCV software, where thresholding based on the h value in hsv space were used. The software algorithm tracked the center of ellipse fitted to the endpoint of a sample cilia-like IPMC structure.



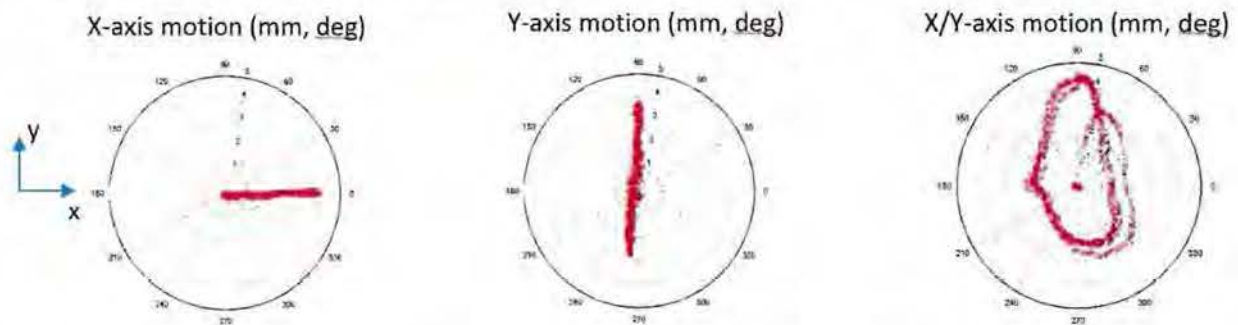
**Figure 1:** Experimental setup for image-based characterization of cilia-like IPMC actuator. The setup consists of an 2D IPMC actuator with colored marker attached to the free end. A digital video camera is used to record the motion of the IPMC actuator and marker. A custom-designed voltage amplifier is used to apply voltages to the electrodes.



Figure 3 shows the experimental results of the image-based characterization approach. The IPMC actuator was first driven with voltages (0 to 3V) to drive the actuator in the x-direction (left plot). Next, the actuator was controlled in the y-direction with +/-3V (middle plot). Finally, the actuator was commanded in 2D (right plot). As shown, the image-based approach was able to capture the motion of the 2D cilia-like actuator and also demonstrates independent control of the actuation in the x- and y-axis, as well as simultaneous 2D motion control. This approach eliminates the need for displacement sensors such as optical laser-based sensors which are often more expensive compared to the image-based approach.



**Figure 2:** IPMC cilia-like fibers used in the image-based characterization experiments.

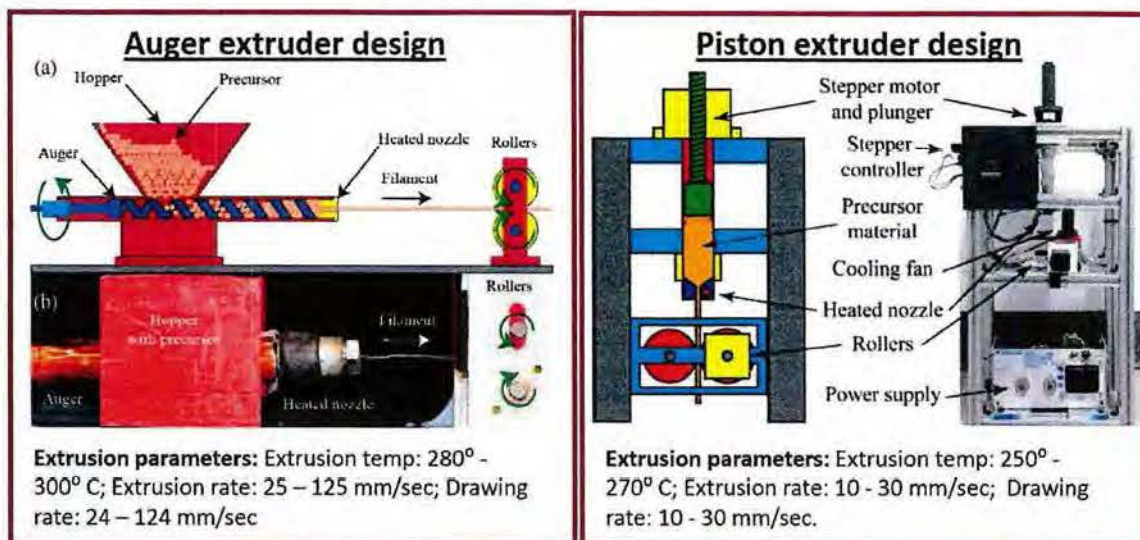


**Figure 3:** Experimental results from image-based motion capture. The IPMC actuator was first driven with voltages (0 to 3V) to drive the actuator in the x-direction (left plot). Next, the actuator was controlled in the y-direction with +/-3V (middle plot). Finally, the actuator was commanded in 2D (right plot).

**Fabrication of various custom-shaped AM structures via additive manufacturing technique:** A new method was developed for fused-filament additive manufacturing (3D printing) of IPMC material to create custom-shaped AM structures, including cilia-like structures. Specifically, a custom 3D printer was created that utilizes custom-made Nafion filament for 3D printing of custom-shaped AM structures.

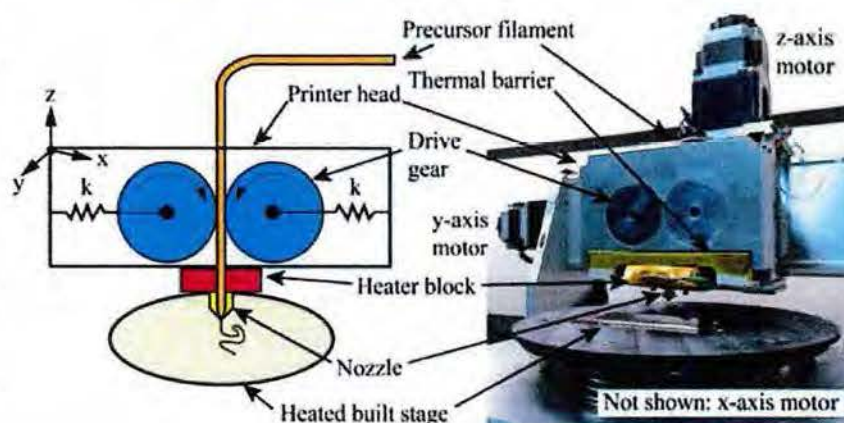
The AM manufacturing process first starts with a solid model of a soft active structure, which can be created in a computer aided design (CAD) software package. Ideally, the body would be a monolithic structure designed with certain sections having actuation capabilities and others with sensing capabilities. Next, the solid model is sent to a custom-designed 3D printer that utilizes an ionomeric precursor filament material to manufacture the fused filament soft polymer structure, layer by layer. Finally, the manufactured component is then chemically “activated” and plated with electrodes to create a fully electroactive body. The AM fabrication technique begins with extruding Nafion filament from commercially obtained Nafion R1100 precursor polymer using a custom designed filament extruder. Figure 4 shows two designs: (a) auger-based design and (b) piston-based design. The piston-based design can operate at a lower temperature range compared to the auger-based design.





**Figure 4:** Filament extruder and example extruded Nafion filament for 3D printing

The custom-design 3D printer for IPMC, shown in Figure 5, consists of a custom-designed x/y/z motion stage, a custom-designed heated print head designed to handle the extruded AM filament, and control software. The custom-designed heater head was specially designed for the relatively soft extruded precursor filament. The feature resolution of the 3D printer

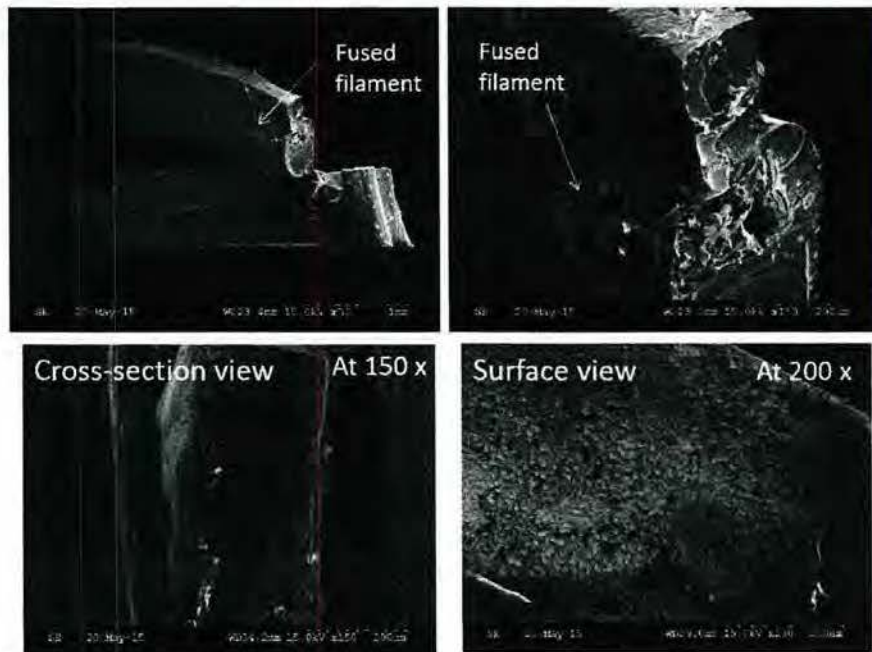


**Figure 5:** Custom-designed 3D printer for printing precursor samples.

is approximately 100  $\mu\text{m}$  and various nozzle diameters can be incorporated. The 3D printer can be used to create small-diameter cilia-like IPMC structures as well as other macro-sized structures. After 3D printing, the precursor samples are then hydrolyzed to "activate" them and subsequently electrodes are applied through an electroless plating process.

SEM images of sample 3D printed IPMC cilia-like structures are shown in Figure 6. The printed samples are more textured than the samples cut from commercially obtained Nafion. Additionally, the printed IPMC is more uneven on one side (the side not against the build stage during the printing process). Like traditional IPMCs fabricated from commercially obtained Nafion sheets, the electrode surfaces exhibit a "mud-cracked" texture caused by drying the IPMCs. It is pointed out that tuning and optimization of the 3D printing step is highly critical and further investigation of this may be needed for optimum printing.





**Figure 6:** SEM images of surfaces of 3D printed IPMC samples.

Various custom-shaped AM structures were fabricated via additive manufacturing technique, as shown in Figure 7, such as thin-membrane-shape actuators and an example linear actuator consisting of layers of printed precursor material. Each layer that was deposited is approximately 0.2 mm.

Thin membrane-shaped actuators



Linear-type actuator



3D printed precursor

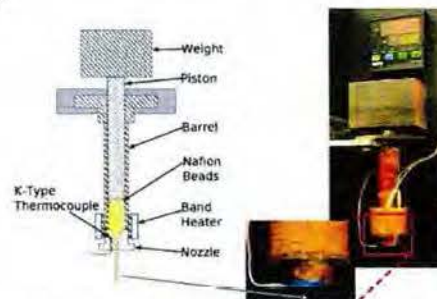


Activated and plated

**Figure 7:** 3D printed IPMC samples: (left) thin membrane-shaped actuators and (middle and right) linear-type actuator consisting of layers of printed precursor material.

**Development of an alternative method of fabricating cylindrical-shaped IPMC actuators:** A simple extruder was fabricated to produce cylindrical Nafion fibers. As shown in Figure 8, the extruder consisted of a weight, piston, barrel, band heater, nozzle, and K-type thermocouple. The extrusion process first place beads of the inactive precursor polymer of Nafion (a polymer which does not contain salt or acid form sulfonyl groups) in the barrel and the piston and weight were inserted above the Nafion to apply a constant pressure to start the extrusion process. The precursor polymer was extruded instead of active Nafion because active Nafion cannot be melt processed, due to the salt groups in active Nafion that impede flow. A hole was drilled in the nozzle which has the same size as the desired diameter of the cylindrical fibers. The band heater and thermocouple were put in contact with the nozzle as shown and were integrated into a controller to regulate the nozzle's temperature. When the polymer was heated above its melting temperature, the piston drove the molten Nafion through the hole. The Nafion fibers were then trimmed from the nozzle when they were the desired length. Inch long Nafion fibers were

obtained through this method with acceptable dimensional consistency. In future work, a fan and a reel could be implemented in the design to allow the extrusion of longer fibers. The fan would be ducted to blow air directly onto the fiber as it emerges from the nozzle to quickly solidify it. The reel would collect the fiber as it is being extruded so that the weight of the fiber does not cause necking. The cylindrical fibers that were extruded using this method had to be activated afterwards. To fabricate smaller diameter fibers, a melt-drawing technique was implemented using the aforementioned setup. In this case, a slight pressure was applied on the piston. Once the tip of polymer filament was out of the nozzle, it was pulled slowly with uniform speed and collected around the rotating drum. The pulling reduces greatly the diameter size of the fiber and induces molecular orientation, increasing the degree of crystallinity and tensile strength of the fiber. The fibers with different dimensions were created by varying the pulling speed and collector distance (Figure 9).



**Figure 8:** Nafion filament extrusion process.



**Figure 9:** Digital optical microscope images of Nafion fibers created with different dimensions using melt-drawing technique in the extrusion process.

Table 1 describes the activation process by which the precursor polymer of Nafion was converted to 'active' salt form Nafion. This process had to be applied to the extruded Nafion fibers before they could be plated. The precursor was treated with a 15% NaOH, 30% DMSO solution at 95 °C for one hour. Then it was treated with an eight molar solution of HNO<sub>3</sub> at 75 °C for three hours. The treatment with the NaOH, DMSO solution converts the polymer to Na salt form Nafion. Treatment with the HNO<sub>3</sub> solution converts it to acid form Nafion. Vigorous agitation must be employed while soaking the Nafion in the NaOH, DMSO solution to prevent the solution from separating.

**Table 1:** Activation process followed by cleaning.

	Procedure	Amount	Temp.	Time
1	Soak in NaOH (15%) DMSO (30%)	300 mL	95 °C	1 hr
2	Soak in 8 M HNO <sub>3</sub>	300 mL	75 °C	3 hr
3	Soak in DI water	300 mL	65 °C	45 min
4	Soak in new DI water	300 mL	65 °C	45 min

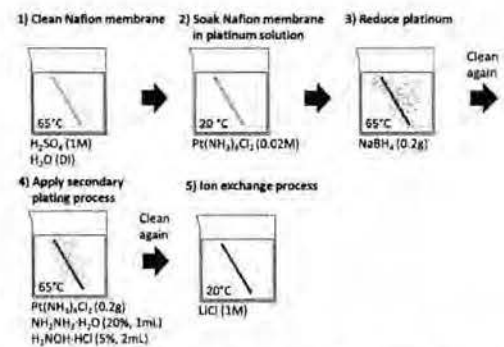
There were five distinct steps involved in plating the Nafion fibers as illustrated in Figure 10. The first step was the initial cleaning process. The second was an ion exchange process, in which platinum cations



replaced the hydrogen cations that were associated with the sulfonyl end groups of the side chains in the surface layers of the Nafion. This was followed with a reduction process by which the surface layers of the fibers that have been impregnated with platinum ions were metalized. The ion exchange process and the reduction process together constitute the primary plating process. In the secondary plating process a reducing agent was applied directly to the platinum salt solution in which the Nafion fiber was immersed. This resulted in platinum being deposited on the surface of the Nafion fiber, adding material to the fiber. Finally, another ion exchange process was conducted in which lithium cations replace the cations associated with the sulfonyl end groups in the core layers of the composite which have not been metalized. Table 2 describes the initial cleaning process. The Nafion was abraded with P1000 grit sandpaper in one direction so that there was a faint grain in the surface of the Nafion parallel to the bending axis of the IPMC. Roughening Nafion in this manner improves the resulting IPMCs actuation characteristics and also increases the surface area of the Nafion for the subsequent processes. The Nafion was then soaked in deionized water for 15 minutes allowing the membrane to hydrate and swell. It was then immersed in a 3% solution of  $\text{H}_2\text{O}_2$  at 65 °C for 45 minutes and soaked again in deionized water for 15 minutes. It was then soaked in a one molar solution of  $\text{H}_2\text{SO}_4$  at 65 °C for 45 minutes. Finally, it was soaked again in deionized water at 65 °C for 45 minutes. The soaking in  $\text{H}_2\text{O}_2$  and  $\text{H}_2\text{SO}_4$  removes most organic matter.

**Table 2: Cleaning Process**

	Procedure	Amount	Temp.	Time
1	Roughen surface with sandpaper	-	room	-
2	Soak in DI water	100 mL	room	15 min
3	Soak in $\text{H}_2\text{O}_2$ (3%)	300 mL	65 °C	45 min
4	Soak in DI water	100 mL	room	15 min
5	Soak in 1 M $\text{H}_2\text{SO}_4$	300 mL	65 °C	45 min
6	Soak in DI water	300 mL	65 °C	45 min



**Figure 10: Plating and ion exchange process.**

Table 3 describes the platinum ion exchange process. The Nafion was then soaked in a 0.02 molar platinum solution. The platinum cations in the solution replace the hydrogen cations that are associated with the sulfonyl groups in the surface layers of the Nafion.

Table 4 describes the reduction process. The Nafion was then removed from the platinum solution and was placed in deionized water that had been heated to 50 °C. 0.2 grams of  $\text{NaBH}_4$  were added to the solution every 30 minutes over the course of three hours. The  $\text{NaBH}_4$  is a reducing agent and metalizes the layers of Nafion that have been permeated with platinum cations. After this process was completed, the Nafion was cleaned by soaking the Nafion in a 0.5 molar solution of  $\text{H}_2\text{SO}_4$  at 65 °C for 45 minutes followed by soaking it in two baths of deionized water at 65 °C for 45 minutes each. Soaking the Nafion in  $\text{H}_2\text{SO}_4$  neutralizes residue from the reduction process. Table 5 describes the secondary plating process. For the secondary plating process, the Nafion was soaked in 300 mL of deionized water at 50 °C to which 0.2 grams of  $\text{Pt}(\text{NH}_3)_4\text{Cl}_2$  has been added. 2 mL of a 5% by volume solution of  $\text{H}_2\text{NOH} \cdot \text{HCl}$  and 1 mL a 20% by volume solution of  $\text{NH}_2\text{NH}_2 \cdot \text{H}_2\text{O}$  were added every 30 minutes to the solution over the course of three hours. This causes platinum to be deposited on the surface of the Nafion. Following this the resulting composite was removed and the resistances across the metalized surfaces were checked. For good conductivity the resistances should be less than 10 Ohms. If they were not, then the secondary plating

process was repeated. After this was completed the composite was cleaned in the same manner as following the primary plating process. Table 6 describes the lithium ion exchange process. Finally, the Nafion fiber was soaked in a one molar LiCl solution at room temperature for 24 hours. The lithium cations in the solution replace the hydrogen cations that are associated with the sulfonyl groups in the core layers of the Nafion. This was done to improve the actuation characteristics of the IPMC. After this process was complete, the IPMC was stored in deionized water.

**Table 3: Platinum Ion Exchange Process**

	Procedure	Amount	Temp.	Time
1	Soak cleaned membrane in 0.02 M Pt Solution with mild stirring	70 mL	room	3-4 hr

**Table 4: Reduction process followed by cleaning**

	Procedure	Amount	Temp.	Time
1	Put in DI water	300 mL	50 °C	-
2	Add NaBH <sub>4</sub> every 30 min and gradually heat to 65 °C	0.2 g	65 °C	3 hr
3	Soak in 0.5 M H <sub>2</sub> SO <sub>4</sub>	300 mL	65 °C	45 min
4	Soak in DI water	300 mL	65 °C	45 min
5	Soak in new DI water	300 mL	65 °C	45 min

**Table 5: Secondary plating process followed by cleaning.**

	Procedure			
1	Add Pt(NH <sub>3</sub> ) <sub>4</sub> Cl <sub>2</sub> to DI water	0.2 g	50 °C	-
2	Soak membrane in solution	300 mL	50 °C	-
3	Add H <sub>2</sub> NOH HCl (5%) every 30 min	2 mL	65 °C	3 hr
4	Add NH <sub>2</sub> NH <sub>2</sub> H <sub>2</sub> O (20%) every 30 min with (3)	1 mL	65 °C	3 hr
5	Check resistance (should be less than 10 Ω)	-	-	-
6	Soak in 0.5 M H <sub>2</sub> SO <sub>4</sub>	300 mL	65 °C	45 min
7	Soak in DI water	300 mL	65 °C	45 min
8	Soak in new DI water	300 mL	65 °C	45 min

**Table 6: Ion exchange process.**

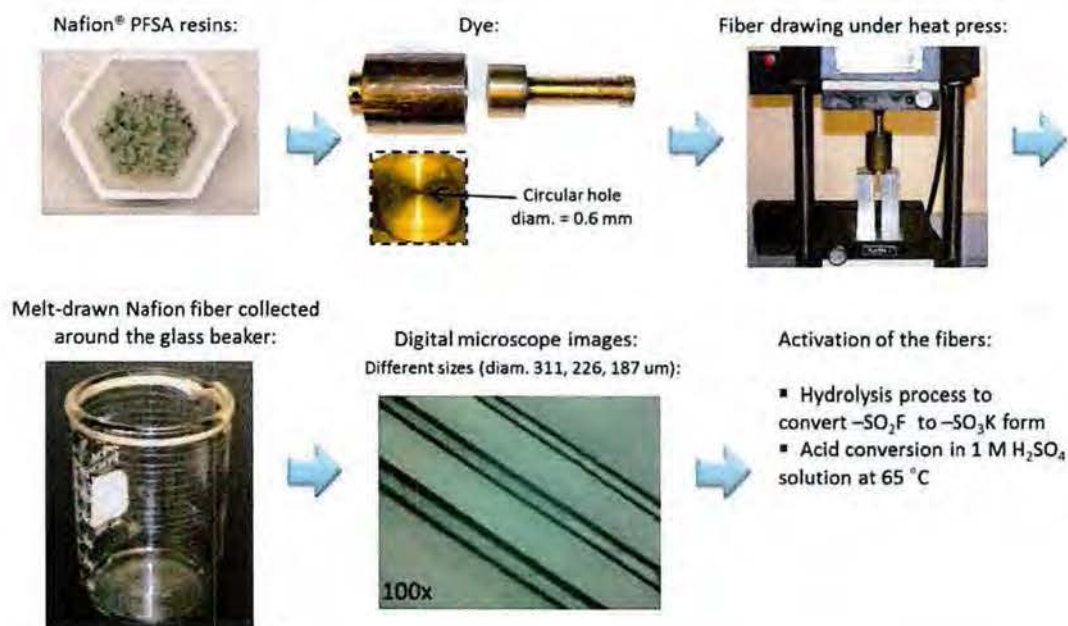
	Process	Amount	Temp.	Time
1.	Soak in 1 M LiCl solution	100 mL	room	24 hr

**Development of small-diameter cylindrical ionomer fibers for AM cilia systems:** The small-diameter AM cilia fibers were fabricated from Nafion precursor resin through a melt-drawing process (Figure 11). First, the inactive Nafion precursor resins in the sulfonyl fluoride (-SO<sub>2</sub>F) form were placed in a custom-made die and preheated. As precursor resin, the Nafion polymer does not have the cation-exchange capability,



but is thermoplastic and can be easily melt-processed into variety of shapes. The preheated mold/die assembly was slightly pressurized initially to generate polymer flow through a die. The exiting polymer thread was pulled with uniform speed and collected around a rotating drum. The pulling (drawing) process reduces greatly the diameter size of the fiber and induces the molecular orientation of polymer chains, increasing the degree of crystallinity and tensile strength of the fiber. The fibers with different dimensions were created by varying the drawing speed and collector distance.

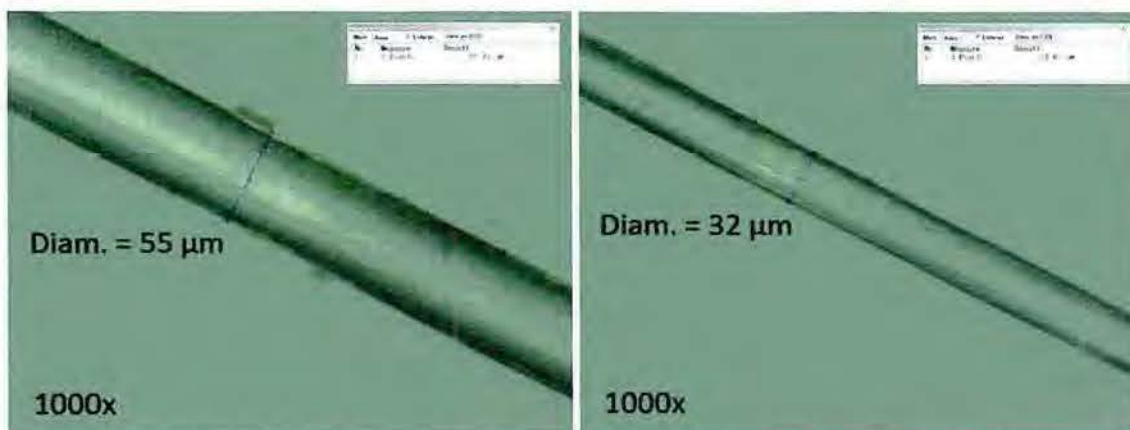
The obtained cylindrical Nafion fibers had to be activated by a chemical treatment to allow the cation-exchange capability. Table 7 describes the activation process by which the Nafion precursor was converted to 'active' sulfonic acid ( $-\text{SO}_3\text{H}$ ) form. The first step was the hydrolysis process in hot aqueous KOH and dimethyl sulfoxide (DMSO) for 1 h to convert the ( $-\text{SO}_2\text{F}$ ) functional groups into sulfonate groups ( $-\text{SO}_3\text{K}$ ). Next, the polymer was converted to sulfonic acid ( $-\text{SO}_3\text{H}$ ) form by treatment in a bath of  $\text{H}_2\text{SO}_4$  solution. Finally, the polymer fibers were cleaned in a bath of de-ionized (DI) water to remove the acid residues.



**Figure 11:** Fabrication of cylindrical AM cilia fibers by melt-drawing process.

**Table 7:** Activation process of ionomer cilia fibers.

	Procedure	Amount	Temp.	Time
1	Soak in KOH (15%), DMSO (30%)	350 mL	95 °C	1 h
2	Soak in $\text{H}_2\text{SO}_4$	350 mL	75 °C	3 h
3	Soak in DI water	350 mL	75 °C	45 min
4	Soak in new DI water	350 mL	75 °C	45 min



**Figure 12:** Digital microscope images of fabricated small-diameter ionomer fibers in different sizes.

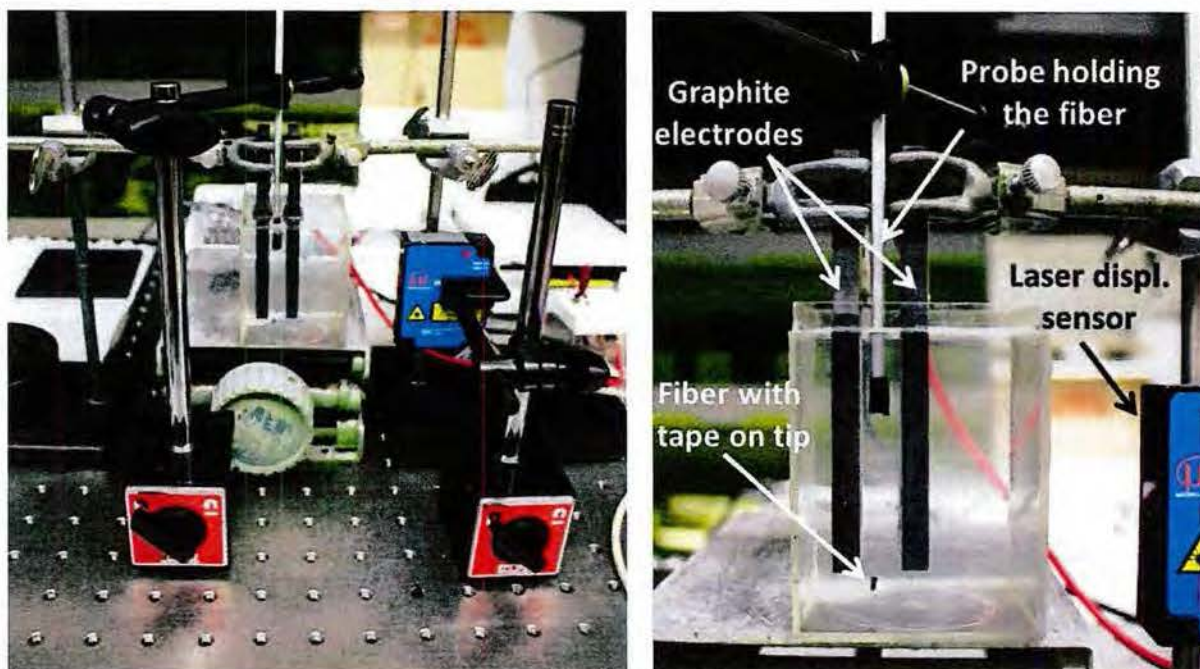
The diameter of melt-drawn fibers is strongly dependent on the dimensions of dye and drawing speed. To down-scale the diameter of Nafion fibers, the dye was down-sized significantly. Table 8 shows the size range of fibers produced with different dyes. With the smallest 175- $\mu\text{m}$  dye, cilia fibers in diameter size of 25–45  $\mu\text{m}$  were successfully produced. Figure 12 shows the fabricated small-diameter ionomer fibers in two different sizes – 32  $\mu\text{m}$  and 55  $\mu\text{m}$ , produced by melt-drawing process using a 175- $\mu\text{m}$  and 210- $\mu\text{m}$  size dye, respectively. The fabricated fibers have a smooth surface morphology and circular cross-section.

**Table 8:** Overall progress in down-scaling the size of cylindrical Nafion fibers.

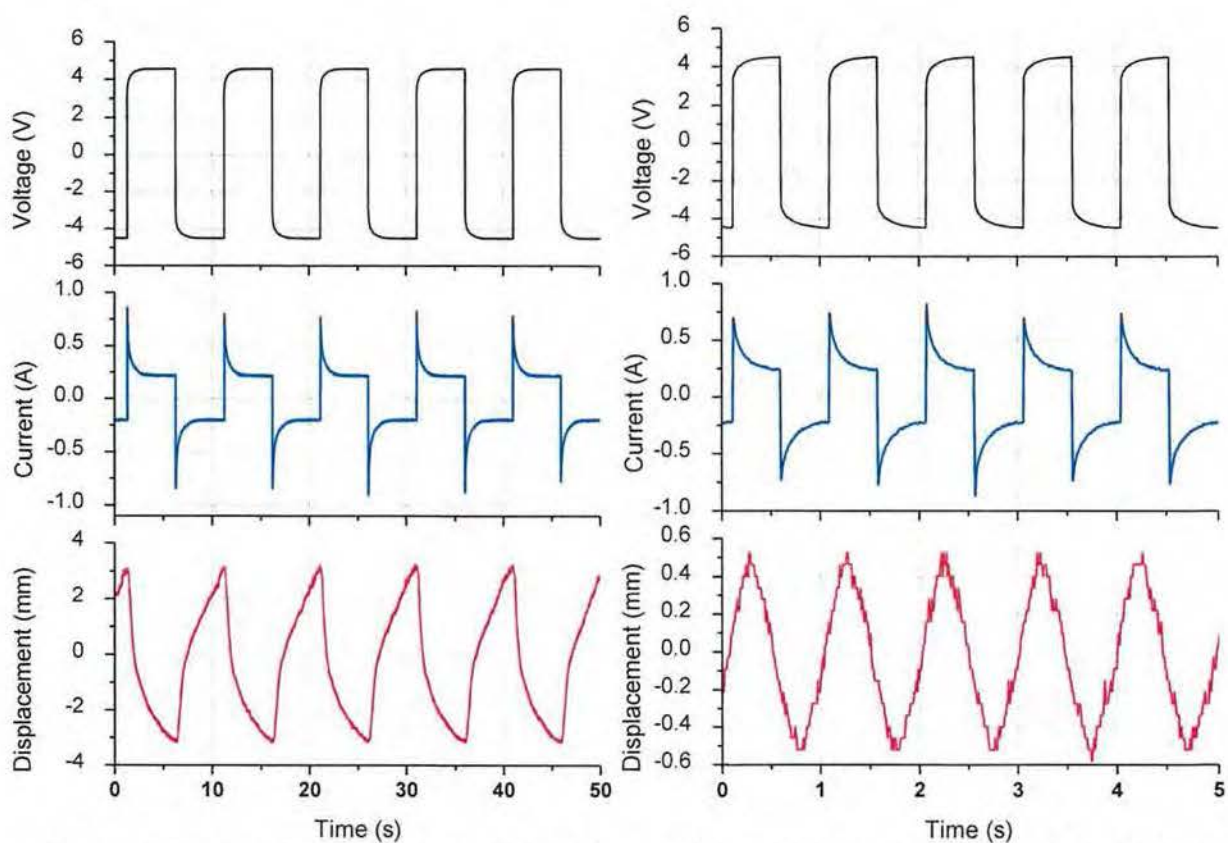
Dye size ( $\mu\text{m}$ )	Fiber diameter range ( $\mu\text{m}$ )
610	200–300
310	70–100
210	30–55
175	25–45

*Characterization of actuation performance of AM cilia fibers under external electric field:* A cylindrical Nafion fiber fabricated by melt-drawing process was suspended in 0.1 M LiCl aq. solution between two parallel graphite electrode plates (Figure 13). The distance between the electrodes was 10 mm and the effective length of the fiber was 42 mm. The bottom end of the fiber reached slightly lower than the electrodes to allow access to the laser beam for displacement measurement. A small piece of tape was attached to the fiber tip, serving as a target for a laser beam. A laser displacement sensor with National Instruments/LabView DAQ system was used for measuring the fiber tip displacement under AC square-wave input of 5 V at frequencies from 0.1 to 1 Hz. Figure 14 shows the measured voltage, current and displacement responses in time for the Nafion fiber under external field of 5 V AC square-wave at 0.1 Hz and 1 Hz. It can be seen that the fiber exhibits repeatable cyclic actuation with adequate response.





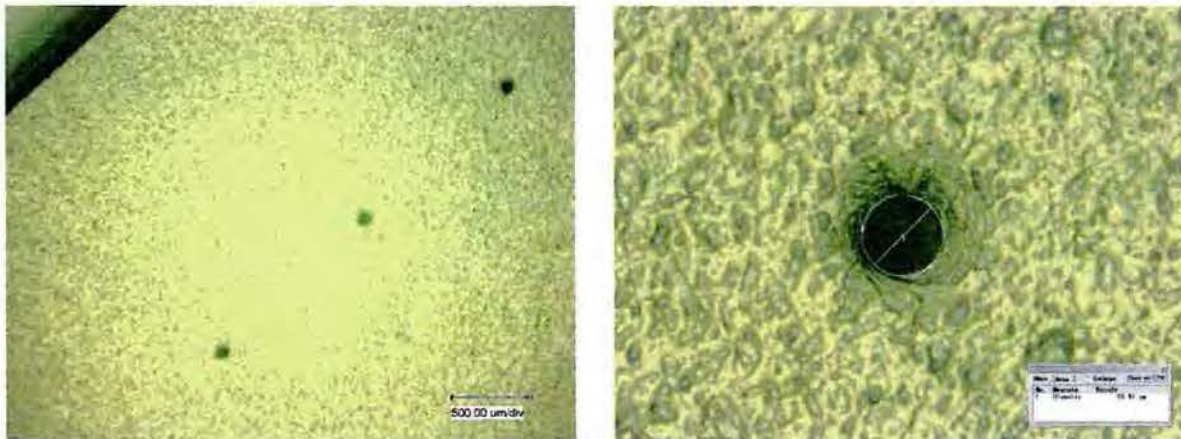
**Figure 13:** Experimental setup used for measuring the displacement of Nafion fiber under external electric field.



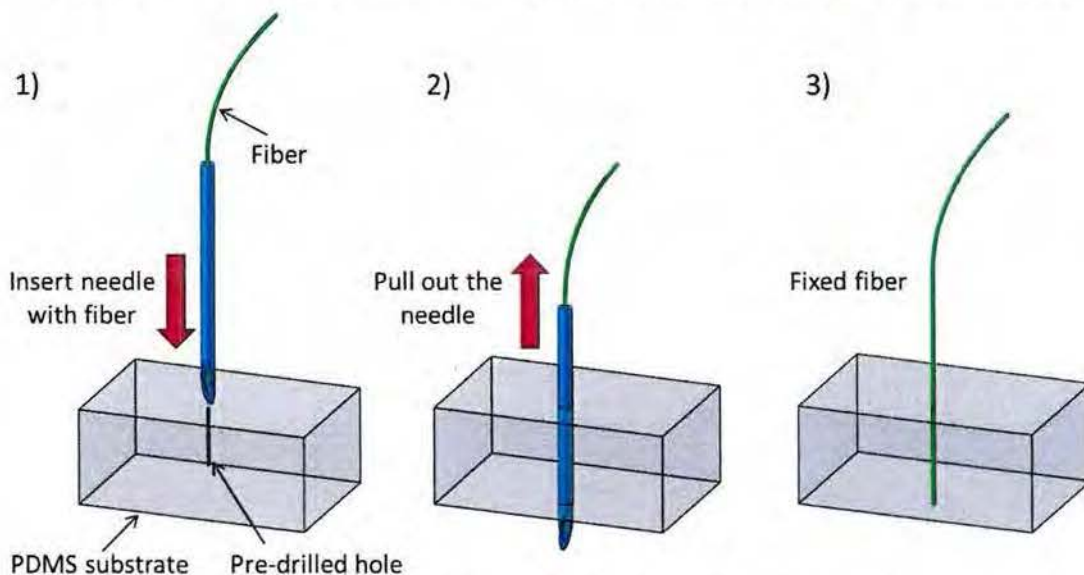
**Figure 14:** Measured voltage, current and displacement responses of cylindrical cilia fiber at 5 V AC square-wave at 0.1 Hz (left) and at 1 Hz (right) in 0.1 M LiCl aq. solution.

**Development of biomimetic AM cilia array system:** The fabricated cylindrical ionomer fibers were used as artificial cilia for designing a prototype cilia array. The biomimetic AM cilia array system was fabricated on a soft and flexible polydimethylsiloxane (PDMS) silicone elastomer substrate. The PDMS substrate was prepared using Sylgard® 184 silicone elastomer kit (Dow Corning). The PDMS pre-polymer mixed at a 10:1 weight ratio of elastomer to hardener was cast into a mold and cured at room temperature for 48 h to obtain a flexible rubber-like substrate layer.

For accurate placement of cilia fibers, an array of holes with diameter of 50  $\mu\text{m}$  was pre-drilled into the PDMS layer using a CNC milling machine (Figure 15). The melt-drawn ionomeric fibers were then planted into the PDMS substrate under a digital microscope using 29-gauge hypodermic needle. The process of fixing cilia fibers in the substrate is illustrated in Figure 16. First, a needle with an ionomer fiber was inserted along the pre-drilled hole and pushed slightly through the substrate. Then, the tip of the fiber was temporarily clamped while pulling out the needle, leaving the ionomer fiber securely fixed in a PDMS substrate. Figure 17 shows the prototype cilia array system consisting of 20 fibers in row with 1 mm spacing in between. The diameter size of the fibers used in this prototype ranges from 70 to 90  $\mu\text{m}$  and free length is 8 mm. The substrate dimensions are 3.6 mm (W) x 2.4 mm (H) x 23 mm (L).



**Figure 15:** PDMS substrate with pre-drilled 50- $\mu\text{m}$  holes for placement of ionomer fibers.



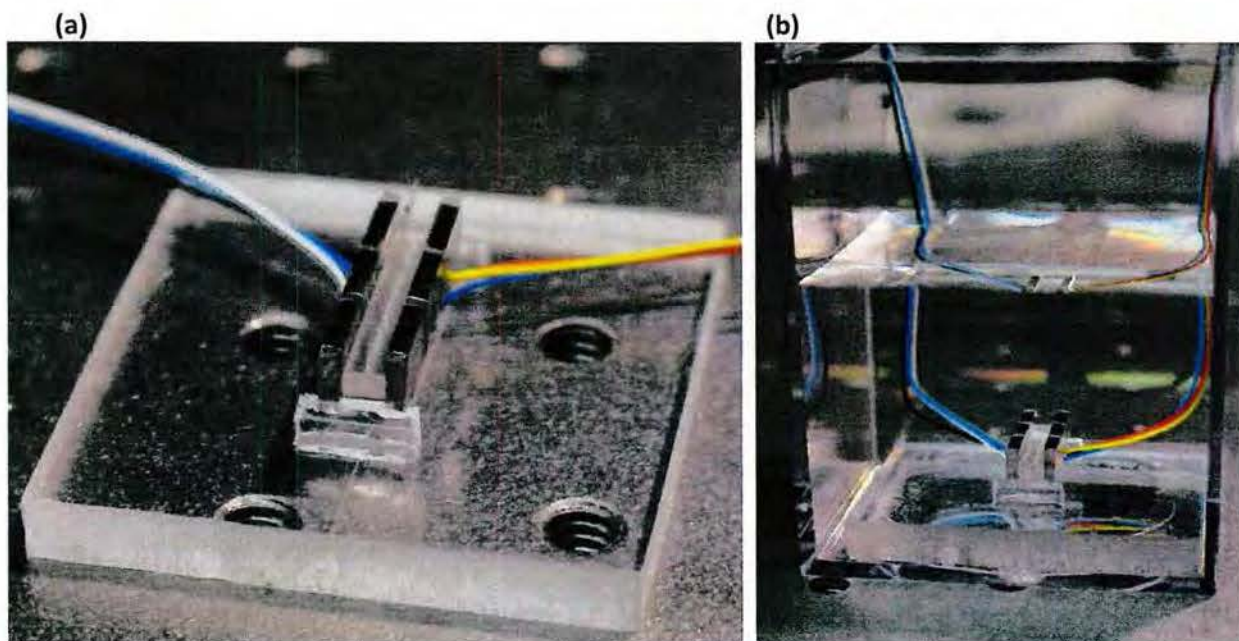
**Figure 16:** Process of fixing the ionomeric cilia fibers into a PDMS substrate.





**Figure 17:** Digital optical microscope images of fabricated prototype AM cilia array (1 x 20, spacing 1 mm).

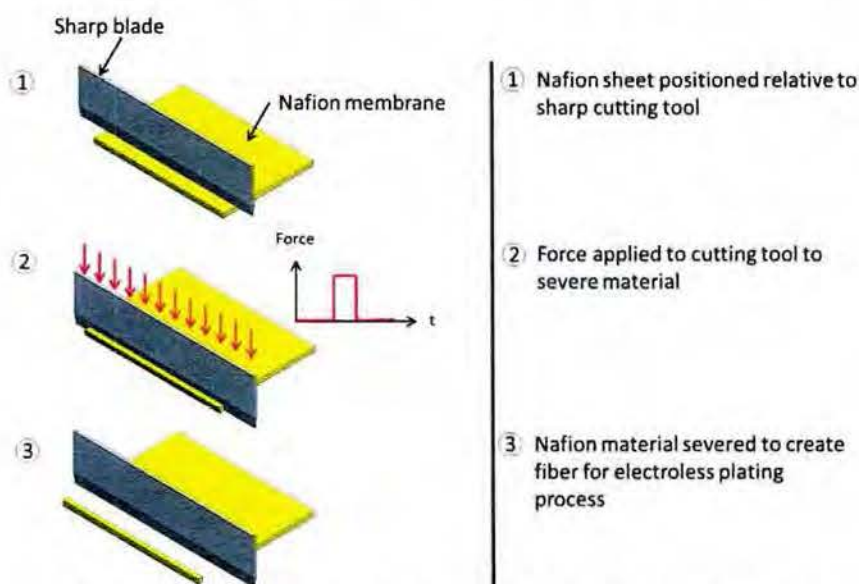
In order to actuate the AM cilia array by an external electric field, a clamp device with 6 external electrodes was designed (Figure 18). The electrode assembly was fabricated from acrylic glass and electrodes were made from Pt foil (thickness 0.001 in). The clamp device consists of 3 sections (pairs) of electrodes that can be used to create a wave-like oscillation of cilia when sections are driven with phase difference. Figure 18(b) shows the prototype cilia fiber (1x20) array mounted between the electrodes and immersed in electrolyte solution.



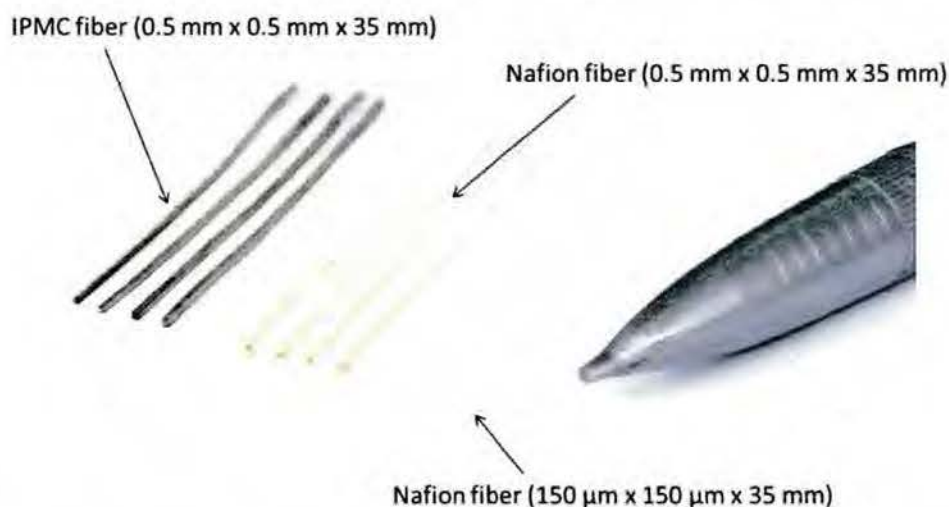
**Figure 18:** (a) Clamp device with 6 external Pt electrodes for actuating the cilia (1x20) array by external field. (b) Cilia array (1x20) mounted between the electrodes and immersed in electrolyte solution.

**Development of square cross-section IPMC Actuators:** Nafion sheet was obtained from a supplier in two conventional thicknesses: 500 and 150  $\mu\text{m}$ . As illustrated in Figure 19, fibers were fabricated from these sheets by positioning the sized Nafion sheet relative to a cutting tool. The sheet was indexed so that the fiber would be cut to a desired width. Generally, the fiber widths were the same as the Nafion sheet

thickness so that the fibers had square cross-sections. A sharp force was applied to the cutting tool severing the Nafion sheet material into small fibers as shown. After the fibers were fabricated, they were plated in a similar manner described above. The cutting process was modified by incorporating so-called slicing technique to further down-size the fiber dimension while maintaining precise control over thickness. In this method, a hydrated dispersion-cast Nafion NRE-212 film with thickness of  $50.4\ \mu\text{m}$  was fixed between two aluminum plates, so that the top edge of the film reached out of the plates (Fig. 21). Several layers of plastic tape (thickness  $\approx 50\ \mu\text{m}$ ) were stacked on the plates on both sides of the film. A Nafion fiber was cut by sliding a sharp blade along the tape. Then, a layer of the tape was removed from both sides for slicing the next fiber with equivalent thickness (Figure 22 and 23). The described slicing technique enables good control over the cut thickness and allows further-down scaling using thinner films.



**Figure 19:** Nafion fiber manufacturing by sheet cutting process.

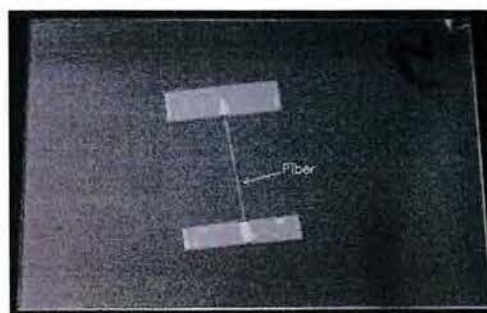


**Figure 20:** From left to right:  $0.5\text{mm} \times 0.5\text{mm} \times 35\ \text{mm}$  IPMC (plated Nafion) fibers,  $0.5\text{mm} \times 0.5\text{mm} \times 35\ \text{mm}$  Nafion fibers,  $150\ \mu\text{m} \times 150\ \mu\text{m} \times 35\ \text{mm}$  Nafion fibers.





**Figure 21:** Nafion fiber fabrication using slicing technique.



**Figure 22:** A sliced Nafion fiber attached on a microscope slide glass. Size:  $54\ \mu\text{m} \times 54\ \mu\text{m} \times 18\ \text{mm}$ .

a) Cross-section view

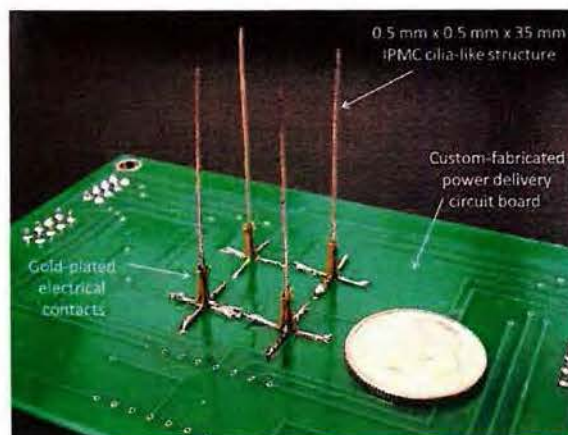


b) Surface view



**Figure 23:** Digital optical microscope images of small-size square cross-section Nafion fibers fabricated using slicing method.

**Development of square cross-section AM fiber cilia array:** The square cross-section IPMCs that were fabricated were used as the artificial cilia in a prototype cilia array. The array consisted of a printed circuit board with  $4 \times m \times n$  independent contacts where  $m$  and  $n$  are the number of IPMC fibers along the length and width of the array respectively. For instance, in the  $2 \times 2$  array shown in Figure 24 there are 16 independent contacts. This allows each IPMC fiber to be driven independently in two dimensions.



**Figure 24:** Cilia board ( $2 \times 2$ ) design.

In summary the accomplishments include: created mm (and also sub-mm)-sized cylindrical AM actuator, created mm-sized hollow tube actuator with integrated sensor, created mm (and also sub-mm)-sized square cross-section AM actuator, developed alternative manufacturing method(s) of AM actuators, created a prototype AM cilia array system ( $D \sim 50$  mm), created a prototype mm-sized AM fiber cilia array ( $2 \times 2$ ) for characterization work, developed image-based characterization method for tracking AM tip displacement, achieved 2D motion for mm-sized AM, and developed physics-based modeling for cylindrical AM actuator.

## VI. Major Problem/Issues

No major problem/issues to report; however, some challenges are noted, such as scaling down presents some challenges, electroplating of small-sized fibers is a challenge, and electrical connection to small AM fibers can be difficult.

## VII. Technology Transfer

The PI, Kwang Kim of UNLV, is collaborating with University of Texas (UT) – Medical School in Houston (Dr. Daniel Kim). Dr. Daniel Kim is a surgeon and established a team of robotists who are working on medical robots.

## X. Foreign Collaborations and Supported Foreign Nationals

N/A.

## IX. Productivity

### A. Refereed Journal Articles

1. K. J. Kim, V. Palmre, T. Stalbaum, T. Hwang, Q. Shen, S. Trabia, **Promising developments in marine applications with artificial muscles: electrodeless artificial-cilia microfibers**, Marine Technology Society Journal, 50.5: 24-34 (2016).
2. Q. Shen, S. Trabia, T. Stalbaum, V. Palmre, K. J. Kim, I. K. Oh, **A multiple-shape memory polymer-metal composite actuator capable of programmable control, creating complex 3D motion of bending, twisting, and oscillation**, Scientific Reports, 6: 24462 (2016).
3. James D. Carrico, Nick W. Traeden, Matteo Aureli, Kam K. Leang, **Fused filament 3D printing of ionic polymer-metal composites (IPMCs)**, Smart Materials and Structures, 24, pp. 125021 (11 pages) (2015).
4. M. A. Tsugawa, V. Palmre, J. D. Carrico, K. J. Kim, K. K. Leang, **Slender tube-shaped and square rod-shaped IPMC actuators with integrated sensing for soft mechatronics**, Meccanic, Vol. 50 (11), pp. 2781-2795 (2015).
5. Q. Shen, V. Palmre, T. Stalbaum, K. J. Kim, **A comprehensive physics-based model encompassing variable surface resistance and underlying physics of ionic polymer-metal composite actuators**, Journal of Applied Physics, 118: 124904 (2015).
6. S. Ruiz, B. Mead, V. Palmre, K. J. Kim, W. Yim, **A cylindrical ionic polymer-metal composite-based robotic catheter platform: modeling, design and control**, Smart Mater. Struct. 24, 015007 (2015).



7. T. Stalbaum, D. Pugal, S. E. Nelson, V. Palmre, K. J. Kim, **Physics-based modeling of mechano-electric transduction of tube-shaped ionic polymer-metal composite**, J. Appl. Phys. 117, 114903 (2015).
8. Q. He, M. Yu, K. J. Kim, Z. Dai, **An ionic electro-active actuator made with graphene film electrode, chitosan and ionic liquid**, Smart Materials and Structures, 24, 065026 (2015).
9. V. Palmre, D. Pugal, K. J. Kim, K. K. Leang, K. Asaka, A. Aabloo, **Nanothorn electrodes for ionic polymer-metal composite artificial muscles**, Scientific Reports 4, 6176 (2014).
10. V. Palmre, S. J. Kim, D. Pugal, K. J. Kim, **Improving electromechanical output of IPMC by high surface area Pd-Pt electrodes and tailored ionomer membrane thickness**, Int. J. of Smart and Nano Materials, 5:2, 99-103 (2014).
11. C. Jo, D. Pugal, I.-K. Oh, K. J. Kim, K. Asaka, **Recent advances in ionic polymer-metal composite actuators and their modeling and applications**, Progress in Polymer Science, 38, 1037-1066, (2013).

#### B. Non-Referred Significant Publications

N/A.

#### C. Books or Chapters

1. K. J. Kim, V. Palmre, D. Pugal, T. Stalbaum, Z. Chen, X. Tan, M. Yamakita, **IPMCs as EAPs: Models**, Polymers and Polymeric Composites: A Reference Series. Electromechanically Active Polymers, F. Carpi, (Ed.), Springer International Publishing AG, (2016).
2. D. Pugal, T. Stalbaum, V. Palmre, K. J. Kim, **Modeling IPMCs with COMSOL: Step-by-Step Guide**, Ionic Polymer Metal Composites (IPMCs): Smart Multi-Functional Materials and Artificial Muscles Volume 2, Shahinpoor, (Ed.): Chapter 5, Royal Society of Chemistry, (2016).
3. James D. Carrico, Maxwell Fleming, Marissa A. Tsugawa, Kam K. Leang, **Precision feedback and feedforward control of ionic polymer-metal composite actuators**, Ionic Polymer Metal Composites (IPMCs): Smart Multi-Functional Materials and Artificial Muscles Volume 2, Shahinpoor, (Ed.): Chapter 11, Royal Society of Chemistry (2016).
4. K. Asaka, K. Kruusamae, K. J. Kim, V. Palmre, K. K. Leang, **IPMCs as EAPs: how to start experimenting with them**, Polymers and Polymeric Composites: A Reference Series. Electromechanically Active Polymers, F. Carpi, (Ed.), Springer International Publishing AG, (2016).
5. K. J. Kim, H. R. Choi, X. Tan, and D. Pugal, **Biomimetic Robotic Artificial Muscles**, World Scientific Publishing Co., (2013).

#### D. Technical Reports

N/A.

#### E. Workshops and Conferences

1. J. D. Carrico and K. K. Leang, **Fused Filament 3D Printing of Ionic Polymer Metal-Composites for Soft Robotic Applications**, 3D printing Workshop, Las Vegas, Nevada, April 15, 2016
2. J. D. Carrico, J. Erickson, K. K. Leang, **Characterization of 3D-printed IPMC Actuators**, SPIE Smart Structures/NDE, Las Vegas, Nevada, March 20-24, 2016, 2016.

3. J. D. Carrico, N. W. Traeden, M. Aureli, K. K. Leang, **Ionic polymer-metal composite fused filament additive manufacturing of soft active structures**, ASME Conference on Smart Materials, Adaptive Structures and Intelligent Systems (SMASIS), September 21-23, 2015 in Colorado Springs, Colorado, 2015, (Jim Carrico and Nick Traeden were winners of the 2015 SMASIS Conf. Best Student Paper Award)
4. K. J. Kim, T. Stalbaum, Q. Shen, October 2016, **Ion concentration and electromechanical actuation simulations of ionic polymer-metal composites**, COMSOL Conference 2016 Boston.
5. S. Trabia, Q. Shen, T. Stalbaum, R. Hunt, T. Hwang, K. J. Kim, August 2016, **Numerical and experimental investigation of a biomimetic robotic jellyfish actuated by ionic polymer-metal composite**, 13th International Conference on Ubiquitous Robots and Ambient Intelligence URAI, IEEE.
6. T. Hwang, V. Palmre, T. Stalbaum, Q. Shen, S. Trabia, K. J. Kim, March 2016, **IPMC cilia system for artificial muscle applications**, Proc. SPIE 979818.
7. T. Stalbaum, S. Trabia, Q. Shen, K. J. Kim, March 2016, **Fluid flow sensing and control using ionic polymer-metal composites**, Proc. SPIE 9782E.
8. Q. Shen, V. Palmre, T. Stalbaum, and K. J. Kim, March 2015, **Comprehensive modeling of ionic polymer-metal composite actuators based upon variable surface resistance and underlying physics of the polymer membrane**, Proceedings SPIE Smart Materials and Structures, 9430-91.
9. T. Stalbaum, S. E. Nelson, V. Palmre, K. J. Kim, March 2015, **Theoretical investigation of ionic effects in actuation and sensing of IPMCs of various geometries**, Proc. SPIE 9432-32.
10. T. Stalbaum, S. E. Nelson, V. Palmre, and K. J. Kim, March 2014, **Multi degree of freedom IPMC sensor**, Proceedings SPIE Smart Materials and Structures, 9056-92.
11. M. Tsugawa and K. J. Kim and K. K. Leang, September 16-18, 2013, **A sectored tube-shaped ionic polymer-metal composite actuator with integrated sensor**, Proceedings of ASME Conference on Smart Materials, Adaptive Structures and Intelligent Systems, Snowbird, Utah.
12. K. Leang, August 26-30, **Precision control of ionic polymer-metal composite actuators**, 7th World Congress on Biomimetics, Artificial Muscles and Nano-Bio (BAMN2013), Jeju Island, South Korea, 2013 (invited presentation).
13. K. J. Kim, June 2013, **Recent Progress in IPMC**, EuroEAP 2013, Zürich, Switzerland (invited talk).
14. K. J. Kim, Oct 29–31, 2013, **Ionic Polymer-Metal Composites: background, applications, manufacturing and modeling**, ESNAM Training School, Cartagena, Spain (invited lecture).

#### F. Patents

N/A.

#### G. Awards/Honors

1. Tyler Stalbaum, PhD student, was selected as an outstanding graduate of UNLV in December 2016.
2. Kwang Kim (the PI) received the 2016 Barrick Distinguished Scholar Award in 2016.
3. Jim Carrico and Nick Traeden were winners of the 2015 SMASIS Conf. Best Student Paper Award for paper: J. D. Carrico, N. W. Traeden, M. Aureli, K. K. Leang, **Ionic polymer-metal composite fused filament additive manufacturing of soft active structures**, ASME Conference on Smart Materials, Adaptive Structures and Intelligent Systems (SMASIS), September 21-23, 2015 in Colorado Springs, Colorado, 2015
4. Kwang Kim (the PI) received Nevada Board of Regents Researcher Award in 2015.
5. Kwang Kim (the PI) was selected as a Collaborative Research and Education (CoRE) Fellow of the UNLV in Spring 2014.



6. Kwang Kim (the PI) was a co-general conference chair of *the 7th World Congress on Biomimetics, Artificial Muscles and Nano-Bio (BAMN2013)*, Jeju Island, South Korea, 2013.
7. Kam K. Leang (co-PI) received Nevada Board of Regents Rising Researcher Award in 2014.
8. Kam K. Leang (co-PI) received UNR College of Engineering Faculty Excellence Award in 2013.

#### **X. Award Participants**

1. Kwang J. Kim, Principal Investigator, UNLV
2. Kam K. Leang, Co-Principal Investigator, UU
3. Shelby Nelson, MS student, UNLV
4. Tyler Stalbaum, PhD student, UNLV
5. Viljar Palmre, Postdoc/visiting scholar, UNLV
6. James Carrico, Ph.D. student, UNR/UU
7. Andrew Pamp, Ph.D. student, UU
8. Nick Traeden, MS student, UU
9. Raeleigh Jones, undergraduate student, UU
10. John Erickson, undergraduate student, UU
11. Marissa Tsugawa, MS student, UNR